Effective Thermal Conductivity of h-BN Filled Epoxy Composites

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Submitted by

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C E R T I F I C A T E

This is to certify that the work in this thesis entitled Effective Thermal Conductivity of h-BN Filled Epoxy Composites by ***Chandan Kumar Jha*** *has been carried out under my supervision in partial fulfilment of the requirements for the degree of Bachelor of Technology in Mechanical Engineering during session 2014 - 2015 in the Department of Mechanical Engineering, National Institute of Technology, Rourkela.*

To the best of my knowledge, this work has not been submitted to any other University/Institute for the award of any degree or diploma.

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ABSTRACT

An optimum solution of heat dissipation has been a consistent requirement after development of electronics devices. These require materials which possesses high thermal conductivity as well as low dielectric constant. Development of polymer composite with desired characteristics could be the solution this problem. In his work it has been tried to study enhancement of the effective thermal conductivity of epoxy polymer composite if h-BN powder is added in it in different volume fraction. Initially a mathematical model was developed and correlations were established to measure effective thermal conductivity of polymer composites for number of volume fractions of particulate added. Further number of epoxy polymer composites reinforced with h-BN filler material was prepared by conventional hand lay-up method in which filler content ranged from 0% to 28.18%. Subsequently effective thermal conductivity of prepared composites were measured by *Unitherm^{TM} Model 2022* in accord with ASTM-E1530 standard. Analytical study shows that after 26 % volume percentage there is a sharp enhancement in effective thermal conductivity of composite. This sudden rise in effective thermal conductivity is due to the overlap between filler particle which leads to the formation of conductive chain. The volume percentage after which this phenomenon occur is termed as percolation threshold.

Nomenclature

K_{eff} = effective thermal conductivity of composite,

K_m = thermal conductivity of matrix material.

K_f = thermal conductivity of filler material.

ϕ = volume fraction of filler material.

R = total thermal resistance of cube.

R_1, R_2, R_3, R_4, R_5, R_6 = resistance of layer 1 2 3 4 5 and 6 respectively.

a = side length of cube.

r = radius of spherical filler.

PMC = polymer matrix composite.

CMC = ceramic matrix composite.

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Chapter - 1
Introduction

1.1 INTRODUCTION:

Composite material is a step towards the optimisation of material characteristics. Composite material is not a recent idea. There exists number of examples in Nature. The coconut palm leaf is an example of fibre reinforcement composite. An example of Natural fibrous composite is wood. Bones contains short and soft collagen fibre inserted in a mineral matrix.

Composite material is a kind of complex multicomponent multiphase system of two or more component material with different properties made by compounding process. It not only maintains the main characteristics of original component but also shows new characteristics which is not possessed by any one of the original component. Generally a single material do not possess all properties required in their application ,therefore new desirable properties are introduced by combining one material with another without compromising with their earlier advantageous properties. Composite material includes mainly three phase.

(i) Matrix phase (continuous phase): It constitutes most of the content. It acts as dispersion medium.

(ii) Reinforcement phase (scattered phase): It acts as disperse phase and remains surrounded by matrix.

(iii) Interphase: It is an interface between reinforcement phase and matrix phase. Near the interface, there is a complex structure of matrix and reinforcement phase, which is different from both them.

1.2 Classification of composites:

Composite materials are classified on different following basis.

1.2.1 **On the basis of matrix material**:

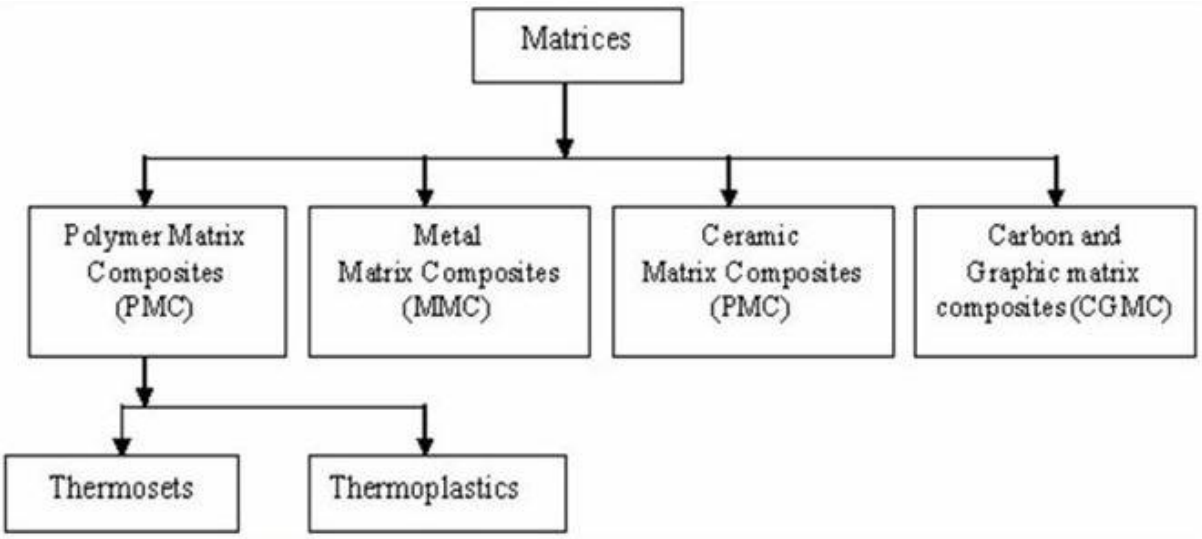

Fig 1.1: classification of composites

(a) Metal matrix composites (MMC'S).

These composites are composed of metal or metal alloys reinforced with particulates or fibres. MMC'S offer high strength, stiffness, creep and fatigue resistance, wear and abrasion resistance and fracture toughness. They are stable at high temperature. Aluminium Titanium and magnesium are mostly used as matrix material.

(b) Ceramic matrix composite (CMC'S).

CMC'S have ceramic as a matrix material like silicates of calcium and aluminium. CMC'S are characterised by their resistance to elevated temperature, good corrosion resistance properties, high melting point, high strength against compressive load, high elasticity modulus and low tensile strain.

(c) Polymer matrix composites (PMC'S).

PMC'S are most commonly used composite among all its counterparts. PMC'S finds wide application in industries because of its easy processability, light weight, resistance to corrosion and desirable mechanical properties. Thermoplastic and thermosets are two main type of polymer.

1.2.2 On the basis of reinforcing material.

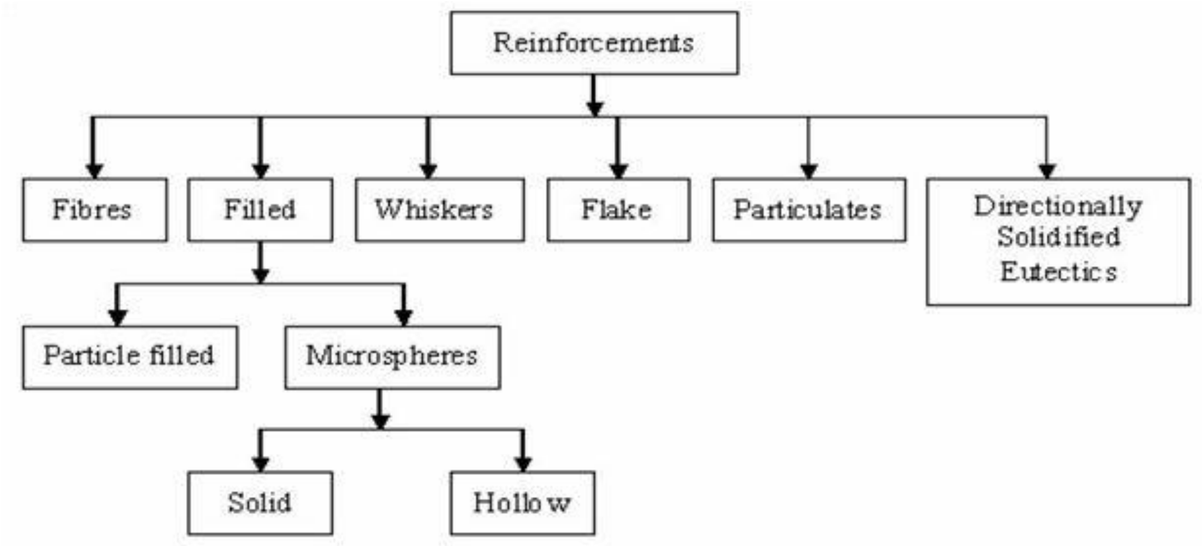

Fig 1.2: Types of reinforcement material

(a) Fibrous fabric, braid reinforced composite material:

Fibres enhance the properties of matrix by transferring the strength to matrix. Mainly used fibres are glass fibres, ceramic fibres, metal fibres etc. composition of the fibre, length, shape, orientation and the mechanical properties of the matrix defines the performance of fibre composite. Orientation of fibre is of great importance as strength is maximum along the longitudinal directional of fibre. That's why it is used in structures subjected to unidirectional load.

(b) Particulate reinforced composite material:

These composites are composed of particulates embedded in matrix body. Particulate can be in the form of flakes or powder. Concrete and wood particle board are Natural examples of this type. Mainly round, square and triangular shapes of particulate are used. Shape size and volume concentration of particulates have significant effect on overall performance of composite. Particulate composites are strengthened by the hydrostatic coercion of fillers in matrices. Particulate reinforcement in three- dimension in composites causes isotropic properties.

Among all types of composite material polymer matrix composite material reinforced with different type of desired filler material is more frequently used as compare to its other counterpart. Polymer composites have also been widely accepted in various industries.

1.3 Application of polymer composite

Polymer composite finds their application in various fields. Based on the requirements like cost, thermal properties, durability, corrosion resistance properties etc different filler materials are added into polymer matrix. Epoxy polymer composite is perhaps the one of the most widely used polymer composite. Epoxy polymer composite provides high strength to weight ratio because of low density of epoxy resin. Because of this property it is used in space industries. One of its most important uses of epoxy polymer composite is in electronic devices. We shall discuss it in detail. Now day demands of electronic devices are continuously increasing. Customers demand devices which are smaller in size as well as provide long duty cycle. This prompts the need of faster and denser circuit. In such a situation effective heat dissipation becomes the major problem in order to avoid disruption in smooth operation of devices and failure of any component of devices. This brings the need of special packaging material which possesses high thermal conductivity, dimensional stability at elevated temperature and good insulator of electricity. A single type material alone is unable to fulfil these entire requirements as they do not possess all required properties. Epoxy polymer composite is one of the solutions of above problem and currently this is being used in electronic industries. Some of the specific uses of epoxy polymer composites in electronic devices are Electronic Packaging, Glob top encapsulation and Printed circuit board. Glop-top is a coating used in chip-on-board assembly. It is composed of special epoxy or resin deposited in the form of drop over a semiconductor chip in order to provide mechanical strength and eliminate contaminants which could lead to disruption of circuit operation. Due to excellent electrical insulating property, high thermal conductivity, low density and easy processabilty of epoxy polymer composites are used in printed circuit boards. It prevents any chance of short circuit.

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Chapter -2

Literature Review

The aim of this chapter is to summarize the background knowledge on the present topic which will be considered in this thesis, and thereby the list the objective of present work. It takes account of review of previous research report on following topic:

1. Particulate Reinforced polymer matrix composites.

2. Thermal Conductivity of Polymer matrix composites.

3. Thermal Conductivity Models.

2.1 Particulate Filled Polymer Matrix Composites

PMC'S have wide acceptance in various industries. These PMC'S are composed of polymers with required filler material as per their application. An improvement in the cost reduction, processing, density control, thermal conductivity, optical effects, control on thermal expansion(for dimensional stability), magnetic properties, electrical properties and mechanical properties such as wear resistance and hardness could be achieved by incorporating particulate filler materials to the polymer matrix. Hard particulate such as metal powder and ceramics and glass fibre are used now a day to improve the wear resistance significantly [1]. Metal particles find their application in manufacturing of electrodes, heaters [2], and composite having high thermal durability at elevated temperature [3]. Ceramic filled polymer composites have attracted researchers greatly in last few decades. Cost reduction and improvement in stiffness are main objectives behind the use of inorganic and non-metallic filler [4, 5].silica filled polymer matrix composite shows improved thermal, mechanical and electrical properties [6, 7]. Many researchers have reported the effect of the size of the particle on the mechanical properties of the composite [8, 9]. A various factor related to fillers such as size, shape, fraction content of particulate by volume affect mechanical properties of the composites significantly.

Mechanical properties such as, tensile and fracture properties and fatigue resistance are significantly affected by structure and shape of silica particles as reported by Yamamoto et al. [10] and Nakamura et al. [11–12]. Study of Moloney et al. [14–16] and Adachi et al. [17] approves the dependency of mechanical properties of epoxy composites on the volume fraction of particles.

2.2 Thermal Conductivity of Polymer Matrix Composites

It has been a general practice to add thermally conductive particulate and fibre into polymers to enhance its effective thermal conductivity. A tremendous amount of works has been done to enhance the thermal properties of particulate filled polymer composites. Most of the works have been experimental. Significant studies have also been done earlier on the transfer of heat through polymer matrix composite. For example- Progelhof et.al [18] presented a thorough study on different methods and models of the thermal conductivity of composite systems. Procter and Solc's [19] studies show thermal conductivity of different types of polymer composites packed with dissimilar fillers and by using Nielsen model their applicability have been confirmed. Tavman [20] investigated the thermal and mechanical properties of poly-ethylene composites packed with copper powder. Study of Mamunya et. Al [21] suggests that there is an enhancement in electrical and thermal conductivity of polymers when metal powders are used as filler material. Weidenfeller et al. [22] investigated the interconnectivity of the disperse particles and its contribution to the thermal conductivity of the polymer composites. There is a significant impact of filler shape factor on thermal conductivity of the composite. This was suggested by Tekce et. al [23].

Polymer materials filled with ceramic powder finds their application in microelectronic since ceramic have high thermal conductivity and low electrical conductivity (electrical insulator). Some important and frequently used ceramic fillers are SiC [25], Si_3N_4 [26], $Sr_2Ce_2Ti_5O_{16}$ [27], CeO_2 [28] AlN [29], Al_2O_3 [30, 32] and ZnO [31]. Above literature suggests that addition of ceramic powder as filler considerably increase the thermal conductivity of composite without any significant increase in electrical properties of composite. Additions of multiple fillers have also been practised to improve thermal conductivity of multi filler filled composite. Apart from experimental studies, numerical method has also been used to estimate the thermal properties of polymer composite [33, 34]. Veyret et al. [33] resorted to numerical method to determine the thermal conductivity of polymer composite.

2.3. Thermal Conductivity Models

A number of theoretical and experimental models and correlation have been developed to determine the thermal conductivity of two phase mixtures and composites.For a two phase composite, the simple approach will be either to arrange the material parallel or in series with respect to direction of heat flow. These two approaches will provide the upper or lower limits of effective thermal conductivity.

For parallel conduction model:

$$K_c = (1-\phi)k_m + \phi k_f \quad 2.1$$

For series conduction model:

$$\frac{1}{k_c} = \frac{(1-\phi)}{k_m} + \frac{\phi}{k_f} \quad 2.2$$

These two equations are derived using Rule of Mixture. Agari and Uno [24] developed a new model and based on this model following correlation was established which take into consideration the mechanism of parallel and series conduction.

$$\log K_c = \phi C_2 \log K_f + (1-\phi)\log(C_1 k_m) \quad 2.3$$

Where, C1, C2 are constants which are determined experimentally. This semi empirical correlation fits well with empirical data, however calculation of constants require ample empirical data for each type of composites. For a highly dilute composite of spherical filler, effective thermal conductivity is given by following exact expression.

$$\frac{k}{k_c} = 1 + \frac{3*(k_d - k_c)}{(k_d + 2*k_c)} \quad 2.4$$

$$\frac{k_{eff}}{K_p} = \frac{k_f + 2k_P + 2\phi_f(k_f - k_p)}{k_f + 2k_P - \phi(k_f - k_p)} \quad 2.5$$

Where

ϕ_f = volume fraction of the dispersed phase;

K = thermal conductivity of the composite.

K_c= thermal conductivity of continuous-phase,

K_p= thermal conductivity of dispersed-phase.

Above equation is called Maxwell equation for dilute composite. For low filler concentrations this model best predict the thermal conductivity, but as the concentration of filler increases there is a formation of conductive chain as particle starts to come in contact with another particle. This conductive chain is directed towards the direction of heat flow. It has been seen that most of the work which has been done towards enhancement of the thermal conductivity is experimental. In the present work we have adopted a new mathematical analytical method to predict the enhancement in thermal conductivity.

2.4 Aim of the present work:

(1) To develop a mathematical model to evaluate effective thermal conductivity of a class of particulate filled polymer composite.

(2) To fabricate the boron nitride filled epoxy composite with two different concentrations by hand lay-out technique.

(3) To experimentally find out the value of effective thermal conductivity of these composites.

(4) To study the effect of incorporation of micro sized boron nitride on the heat conductivity of epoxy.

(5) To validate the theoretical model by comparing the results with measured values.

(6) To identify the potential applications of these composite in microelectronics.

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Chapter -3

MATERIALS AND METHODS

This chapter is dedicated to details of material and method used in present analytical model. Further detail description of experimental procedure used has been presented.

3.1 Material:

Metals, ceramics and polymers are most often used as matrix material for processing of composite. Most commonly used matrix material is polymer matrix since polymers are cheaper, easy to fabricate into complex part with less tooling cost, show excellent properties at room temperature as compare to ceramic and metal matrix. Polymer matrix can be of two type viz. Thermoplastic and thermoset. Due to huge 3D cross link structure thermosets show good electrical insulation properties, outstanding thermal stability and better creep resistance. Epoxy, polyester, vinyl ester and phenolic resin are example of thermosets which are most commonly used. Perhaps epoxy is the one of the most commonly used thermoset resin. Epoxy is used for packaging material since it has low density (1.2 gm. /cc), excellent processability, low dielectric constant, very good electrical insulators (and hence defend electrical components from short circuiting) and low cost. Keeping all above benefits in mind, for present model epoxy has been selected as matrix material. Some common properties of epoxy resin in tabulated below.

Table 3.1: Properties of epoxy

Characteristic Property	Inference
Density	1.1 gm/cc
Compressive strength	90 Mpa
Tensile strength	58 Mpa
Micro-hardness	0.085 Gpa
Thermal conductivity	0.363 W/mK
Glass transition temperature	98^0C
Coefficient of thermal expansion	62.83 ppm/ ^{0}C
Electrical conductivity	0.105 X 10^{16} S/cm

Epoxy is found in both liquid and solid form. Epoxy is formed by step growth polymerisation reaction between biphenol and epichlorohydrin. Thermal conductivity of epoxy alone is very low (0.363W/m. K).

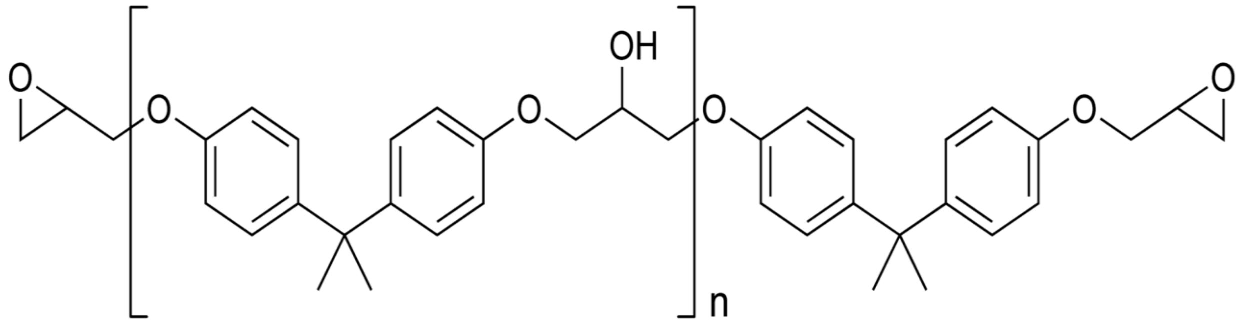


Fig 3.1: epoxy polymer chain

Fig 3.2 : liquid epoxy resin.

3.2 Filler material:

Hexagonal Boron Nitride (hBN) is also sometimes called “White Graphite” has hexagonal crystal structure similar to Graphite, but hBN is much superior to Graphite. Some important characteristics of hBN is listed in table.

Table 3.2: Properties of boron nitride

Characteristic Property	Inference
Chemical formula	BN
colour	White
Density	2.1 gm/cm^3
Melting point	3000^0 C
Coefficient of friction	0.15 to 0.17
Dielectric constant	4 MHz
Dielectric breakdown strength	35 KV/mm
Young's modulus	20-102 MPa
Thermal expansion coefficient at room temperature	1 X 10^{-6} $/^0$C (parallel to press dir.) 4 X 10^{-6} $/^0$C (perpendicular to press dir.)
Thermal conductivity at 293 K	300 W/mK
Temperature stability	1000^0C in Air 1400^0C in Vacuum 1800^0C in inert atmosphere

Fig 3.3: h-BN powder

3.2.1 Key Properties:

1) Thermal conductor (better heat dissipation property).
2) Low dielectric constant.
3) Low thermal expansion.
4) High temperature stability.
5) Electrical insulator.

6) Chemically inert.
7) Non-toxic.
8) Easy machinability.

3.3 Method

3.3.1 Analytical modelling.

A analytical approach has been adopted to describe present model and a correlation has been established for theoretical calculation of effective thermal conductivity of epoxy-hBN composite. Shape of the filler was assumed to be spherical and it was supposed to be evenly distributed in matrix material in FCC arrangement, since FCC is the most effective way to fill spherical filler in matrix. Let side length of cubic matrix is "a" and radius of spherical filler is "r". Following expression was used to evaluate the volume fraction of particulate filler in matrix.

$$\phi = \frac{4\mathrm{x}\frac{4}{3}x\pi r^3}{a3} \qquad 3.1$$

if we put a= 4r we get $\phi = 0.2618$. We have considered three cases:

I. ϕ = 0 (pure matrix material)
II. ϕ = 0.15 (less than 0.2618)
III. ϕ = 0.2618.

Cubic is divided into number similar layers and series of thermal resistance is considered in the direction of heat flow. Total resistance of cube is given by following expression

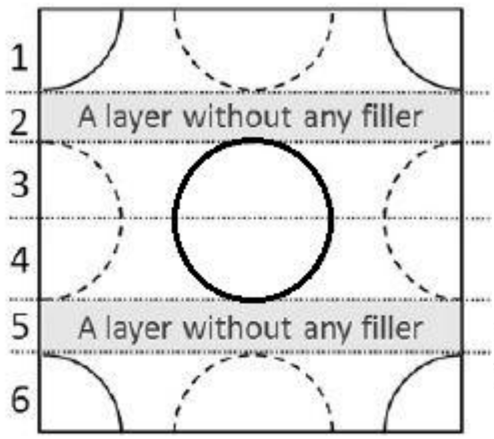
$$R_{total} = \sum_{i=1}^{n} R_i \qquad 3.2$$

Here we have ignored interface resistance between particulate and matrix material to make the analysis simpler.

Case-I : ϕ = 0.0

In this case total resistance of composite will be the resistance of pure matrix material alone.

Case- II : ϕ = 0.15 (less than 0.2618)



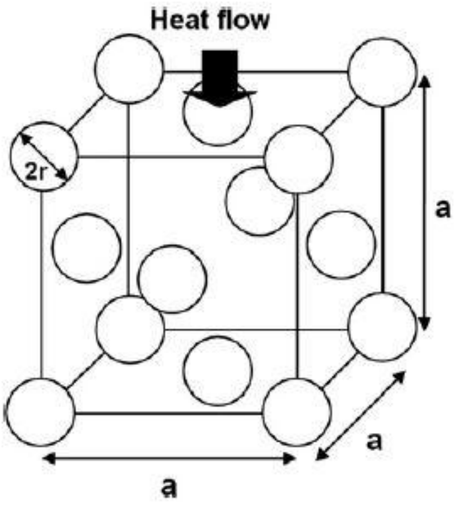


Fig 3.4: side view of cube (ϕ < 0.2618)   fig 3.5: FCC arrangement of filler in matrix

Resistance of layer 1,3 4 and 6 would be same and can be given by following expression.

$$R_1 = R_3 = R_4 = R_6 = \frac{1}{2\pi(k_m - k_f)} \int_0^r \frac{1}{u^2 - y^2}\, dy \qquad 3.3$$

$$R_1 = R_3 = R_4 = R_6 = \frac{1}{4\pi(k_m - k_f)\mathrm{u}} \log_e\left(\frac{u+r}{u-r}\right) \qquad 3.4$$

Similarly resistance of layer 2 and layer 5 would be same

$$R_2 = R_5 = \frac{a-4r}{2K_m a^2} \qquad 3.5$$

Total resistance would be sum of all above resistance. An expression for total resistance for this case is given below.

$$\mathrm{R} = \frac{1}{\pi(k_m - k_f)\mathrm{u}} \log_e\left(\frac{u+r}{u-r}\right) + \frac{a-4r}{K_m a^2} \qquad 3.6$$

Where

$$u = \sqrt{\frac{k_m a^2}{2\pi(k_m - k_f)} + r^2}$$

<u>Case –III</u> **:** ϕ = 0.2618

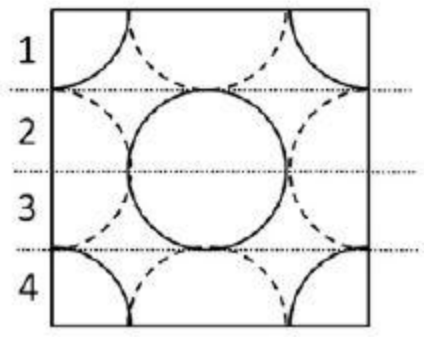


Fig 3.6: side view of cube (ϕ =0.2618).

Here all 4 layers are equivalent and resistance of each layer can be given by following expression.

$$R_1 = R_2 = R_3 = R_4 = \frac{1}{2\pi(k_m - k_f)} \int_0^r \frac{1}{u^2 - y^2}\, dy \tag{3.7}$$

$$R_1 = R_2 = R_3 = R_4 = \frac{1}{4\pi(k_m - k_f)\mathrm{u}} \log_e\left(\frac{u+r}{u-r}\right) \tag{3.8}$$

An expression for total resistance for this case is given below.

$$\mathrm{R} = \frac{1}{\pi(k_m - k_f)\mathrm{u}} \log_e\left(\frac{u+r}{u-r}\right) \tag{3.9}$$

Where

$$u = \sqrt{\frac{k_m a^2}{2\pi(k_m - k_f)} + r^2}$$

Now, k_{eff} for all above case was found out using following expression

$$k_{eff} = \frac{a}{Ra^2} = \frac{1}{Ra} \tag{3.10}$$

3.3.2 **Experimental procedure:**

A very simple of composite processing called Hand lay-up technique has been used to prepare physical model. The processing steps are very simple.

- Firstly, a mould release spray (heavy duty silicon spray) is sprayed on inner surface of mould in order to prevent sticking of composite to the surface.

- Appropriate amount of epoxy and h-BN powder was weighted on weighting machine carefully.

- Weighted epoxy and h-BN were mixed together into mould and then required amount of hardener was added drop by drop.

- Composite having two different volume fraction 15% and 26.18% of filler were prepared.

- After uniform mixing mould was left at room temperature for next 24 hour after which moulds were smashed and composite were taken out.

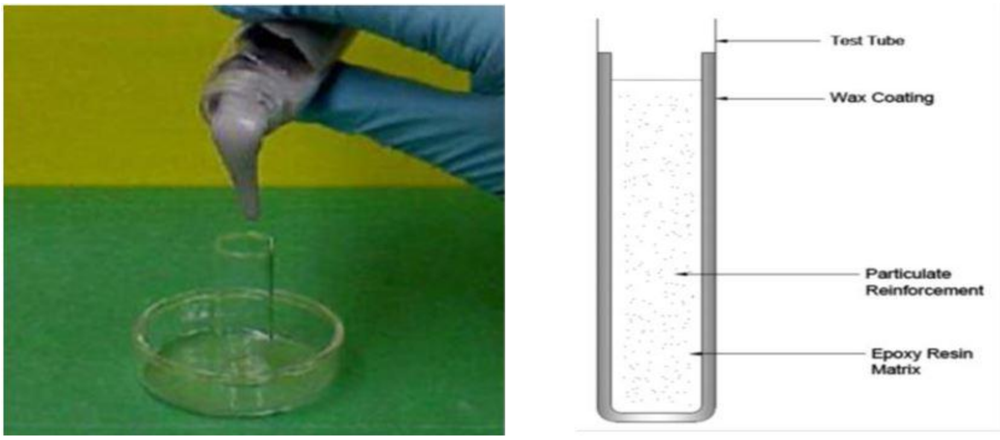


Fig 3.7: fabrication of composite by hand lay-up method

3.4 Experimental Determination of Thermal Conductivity

Effective thermal conductivity measurement of composite was done using Unitherm^{TM} model 2022 tester. **ASTME-1530** standard is followed for this measurement.

3.4.1 **Operating principal of Unitherm^{TM} model 2022 tester.**

Test sample is held between two polished surfaced and compressive force applied to avoid any layer of air at interfaces. Temperatures different is applied across the test specimen. Heat flows from top, passes through the length of sample to bottom, and hence a temperature gradient is established along the length of the test specimen. Once the steady state is achieved temperature drop across the test sample is measured by temperature sensor. Thermal conductivity is then obtained using following expressions.

Fig 3.8: Unitherm^{TM} model 2022 tester

$$Q = \frac{k*A*(T_1 - T_2)}{x} \qquad 3.11$$

$$R = \frac{x}{(K*A)} \qquad 3.12$$

$$K = \frac{x}{(R*A)} \qquad 3.11$$

Fig 3.9: final fabricated epoxy-hBN

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Chapter -4

Results and Conclusions

This chapter presents the results of model analysis. Effective thermal conductivity of epoxy-hBN composite obtained from theoretical model analysis and that obtained from conducted experiment is shown here. Further difference between the results obtained from both above method will be discussed and we will try to find out causes of this difference.

4.1 Effective thermal conductivity of epoxy-hBN composite obtained from theoretical analysis and experimental measurement.

Effective thermal conductivity of epoxy-hBN composite for different volume percentage of filler content is shown below in bar chart.

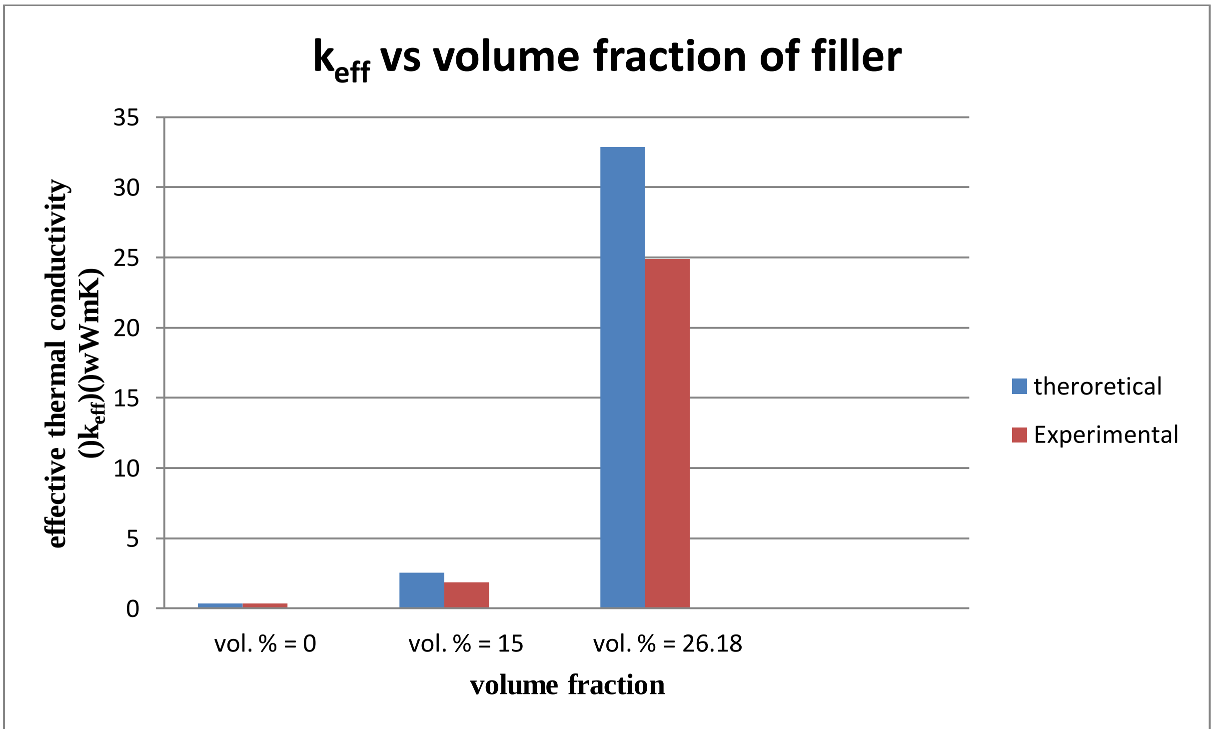

Fig 4.1: A bar chart showing experimental and theoretical result

4.2 Discussion

The following observation can be noticed from above bar chart

(a) There is a sharp increase in effective thermal conductivity of composite when volume fraction of particulate is 26.18 %. This sharp enhancement in thermal conductivity can be explained by the fact that after 26.18% volume fraction of particulate in matrix material, particulates start overlapping with each other which lead to the formation of conductive chain in heat flow direction which eventually results in sharp enhancement in thermal conductivity.

(b) There is a significant difference in result obtained from analytical modelling and that obtained from experimental measurement. This difference is due to the fact that we neglected the interfacial resistance between the contact surfaces of particulate and matrix material. This difference is also evident that interfacial resistance have significant contribution in total thermal resistance and hence cannot be safely neglected.

4.3 Conclusions:

Following conclusion has been drawn from theoretical analysis and experimental investigation.

(i) Successful manufacturing of epoxy-hBN polymer composite is possible by conventional Hand - layup method

(ii) The expressions which have been developed in present work can be used to determine the effective thermal conductivity of composite material with different volume fractions.

(iii) The magnitude of effective thermal conductivity obtained from analytical model and that obtained from experimental investigation for various volume fractions are under agreement for volume percentage of particulate ranging from 0 to 26.18%.

(iv) Inclusion of h-BN powder in epoxy polymer composite results in substantial increase in effective thermal conductivity of epoxy-hBN composite. For inclusion of 15 % of h-BN by volume, effective thermal conductivity rises by 4.16%. Similarly with the inclusion of

26.18 % of h-BN by volume, corresponding increment in the effective thermal conductivity is found by 67.57 %.

(v) This new developed epoxy-hBN composite can be employed for various applications like electronic packaging, glob top encapsulation, printed circuit board etc.

5.3 Scope for Future work:

In the present work effect of volume fraction of particulate has been studied theoretically and experimentally. Apart from volume fraction there are other parameters which can affect the thermal conductivity positively, such as

(1) Effect of size and shape of particulate can be investigated.

(2) Instead of particulate, fibre or nanoparticle can be used as filler material and effect of this change can be studied.

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